

DOCKET NO: 257253US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
BERND ZASCHKE, ET AL. : EXAMINER: COONEY, J. M.  
SERIAL NO: 10/507,315 :  
FILED: SEPTEMBER 10, 2004 : GROUP ART UNIT: 1796  
FOR: GRAFT POLYOLS WITH A :  
BIMODAL PARTICLE SIZE  
DISTRIBUTION AND METHOD FOR  
PRODUCING GRAFT POLYOLS OF THIS  
TYPE, IN ADDITION TO THE USE  
THEREOF FOR PRODUCING  
POLYURETHANES

APPEAL BRIEF

SIR:

The following is an appeal of the examiner's final rejection of June 10, 2008 of claims 1, 2 and 4-16 as obvious. A Notice of Appeal along with a petition for a one-month extension of time was filed on October 10, 2008. A petition of a two-month extension of time is submitted herewith.

(i) Real Party In Interest

The real party in interest is BASF, AG, now BASF, SE, by assignment recorded at reel/frame 015932/0176.

(ii) Related Appeals And Interferences

Appellants, appellants' legal representative and the assignee are not aware of any related appeals and interferences which will directly affect or be directly affected, or have a

bearing on the Board's decision in the pending appeal. Any copies of decisions rendered by a court or the Board, if any, in any proceeding identified would be attached as related proceedings appendix (x).

(iii) Status Of Claims

Appellants' state the status of all the claims in the proceeding as follows:

Claims 1, 2 and 4-16 are rejected and active in this application and are herein appealed.

Claims 3-5 and 17 have been canceled.

No claims have been identified as allowed or confirmed.

No claims have been identified as withdrawn.

No claims have been identified as objected to.

(iv) Status Of Amendments

The advisory action of September 4, 2008 states that appellants' amendment of August 19, 2008 will be entered for the purposes of appeal. The status of such amendment is, for purposes of appeal, entered. A copy of the claims herein appealed, as amended on August 19, 2008, is attached as appendix (viii).

(v) Summary Of Claimed Subject Matter

The claimed invention is directed to a graft polyol comprising (page 5, lines 3-6)  
small particles having a particle diameter of from 0.05 to 0.7  $\mu\text{m}$  and (page 5, lines 6-7)  
large particles having a particle diameter of 0.4 to 5.0  $\mu\text{m}$ , wherein said large particles have a larger particle size than said small particles, and (page 5, lines 7-8)  
wherein the graft polyol has a bimodal particle size distribution, (page 5, lines 4-5)

the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap, and (page 5, lines 9-10 and page 7, lines 29-32)

the graft polyol has a total solids content of from 5 to 65% by weight (page 5, lines 5-6)

wherein the total content of the solids consists of a volume fraction of from 5 to 45% of the small particles and a volume fraction of from 95 to 55% of the large particles, wherein the volume fractions of the small and large particles sum up to 100%. (page 5, lines 11-13)(claim 1)

The claimed invention is also directed to a process for the preparation of a graft polyol having a bimodal particle size distribution comprising (page 5, lines 15-18)

small particles having a particle diameter of from 0.05 to 0.7  $\mu\text{m}$  and (page 5, lines 6-7)

large particles having a particle diameter of 0.4 to 5.0  $\mu\text{m}$ , wherein said large particles have a larger particle size than said small particles, and (page 5, lines 7-8)

wherein the graft polyol has a bimodal particle size distribution, (page 5, lines 4-5)

the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap, and (page 5, lines 9-10 and page 7, lines 29-32)

the graft polyol has a total solids content of from 5 to 65% by weight (page 5, lines 5-6)

wherein the total content of the solids consists of a volume fraction of from 5 to 45% of the small particles and a volume fraction of from 95 to 55% of the large particles, wherein the volume fractions of the small and large particles sum up to 100% (page 5, lines 11-13)

comprising preparing graft polyols in a semibatch process, (page 5, lines 28-30)  
wherein the initially taken reaction mixture contains in each case at least one carrier polyol, a macromer and a graft polyol having a monomodal particle size distribution, (page 5, lines 30-32) more than 3% by weight of the solids content in the resulting graft polyol consisting of the solids content of the graft polyol used in the initially taken reaction mixture and having a monomodal particle size distribution, (page 5, lines 33-36) and the amount of the macromer used in the initially taken reaction mixture is from 1 to 30% by weight, based on the total weight of the ethylenically unsaturated monomers wherein the amount is sufficient to form small particles in the further course of the reaction. (page 5, lines 36-41) (claim 11)

The claimed invention is also directed to a process for the preparation of polyurethanes comprising (page 5, lines 43-45)

reacting (a) organic and/or modified organic polyisocyanates with (b) graft polyols and, optionally (c) additional compounds having hydrogen atoms reactive toward isocyanates, in the presence of (d) catalysts, (e) optionally water and/or other blowing agents and, optionally (f) additional assistants and additives, (page 5, line 45 through page 6, line 3)

wherein the (b) graft polyols have a bimodal particle size distribution (page 5, lines 4-5) and a total solids content of from 5 to 65% by weight (page 5, lines 5-6), which comprises small particles having a diameter of from 0.05 to 0.7  $\mu\text{m}$  (page 5, lines 6-7) and large particles having a diameter of from 0.4 to 5.0  $\mu\text{m}$  wherein said large particles have a larger particle size than said small particles, (page 5, lines 7-8)

the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap, (page 5, lines 9-10 and page 7, lines 29-32)

and the total content of solids consists of a volume fraction of from 5 to 45% of the small particles and a volume fraction of from 95 to 55% of the large particles, wherein the

volume fractions of the small and large particles sum up to 100%. (page 5, lines 11-13)  
(claim 16)

Claims 1, 11 and 16 are the only independent claims involved in this appeal, whose subject matter are defined above.

Graft polyols have been used in the preparation of polyurethane foams to increase the hardness of the resulting polyurethane foam. Graft polyol addition can have an adverse effect on cell opening behavior and flow behavior in the foam mold such that graft polyol containing polyurethanes having good cell opening behavior and flow behavior are sought.

The claimed invention addresses the problem by providing a graft polyol comprising small particles and large particles having **a bimodal particle size distribution**. Appellants have discovered that a graft polyol having a bimodal particle size distribution in which the **peaks do not overlap** having a small particle and large particle distribution as claimed, provides for advantageous properties when incorporated into a polyurethane composition. Such a graft polyol is nowhere disclosed or suggested in the cited art of record.

No means plus function or step plus function as permitted by 35 U.S.C. 112, sixth paragraph are used and therefore none are identified.

(vi) Grounds Of Rejection To Be Reviewed On Appeal

1) The rejection of 1, 2 and 4-17 under 35 U.S.C. § 103(a) over EP 786,480 in view of Perry et al. U.S. 6,127,443 is presented for review.

2) The rejection of claims 1, 2 and 4-17 under 35 U.S.C. 112, second paragraph is presented for review.

(vii) Argument

The examiner has committed reversible error in concluding the claimed invention to be indefinite as the metes and bounds of a graft polyol having **a bimodal particle size distribution and the peaks of the large and small particles...do not overlap** is clear to those of ordinary skill in the art.

The examiner has erroneously concluded that claims 1, 2 and 4-17 are indefinite since the “degree of overlap” is unable to be determined and “determination of the beginning and end of a ‘peak’ is a subjective determination” and therefore the examiner is unable to determine what distribution of particle sizes are included or excluded by the claims.

The metes and bounds of the term “bimodal size distribution” and “peaks...do not overlap” are clear to those of ordinary skill in the art.

*Opinion Evidence By Experienced Polyurethane Researcher Unrebutted By Examiner*

The examiner has erroneously dismissed the declarations of February 26, 2008 and August 19, 2008, by Dr. Daniel Freidank, a researcher for BASF, the assignee of the above-identified application.

Dr. Freidank has been a researcher in the field of polyurethane research and development since 2003. Dr. Freidank has also supplied an evidentiary declaration on July 26, 2007.

Dr. Freidank provides **his opinion**, that the phrase “that the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap” means that the data measured between the peaks are below the background-noise, or, with other words, between the peaks is an area of measurements not larger than the background-noise. This is the opinion of a researcher who has been conducting polyurethane research since 2003.

Dr. Freidank further opines that polarization intensity differential scattering is an established method for determining the particle size in the range of the graft polyols claimed and that the Laser Diffraction Particle Size Analyzer LS 230 is commonly used for these measurements. Again, this is the opinion of a researcher who has been conducting polyurethane research since 2003.

**Dr. Freidank concludes** in the penultimate paragraph on page 2 of his declaration that the phrase “that the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap” **is clear for the person skilled in the art.**

Thus, appellants have provided **evidence** in the form of the opinion declaration of Dr. Daniel Freidank that the claim term “that the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap” is clear for the person skilled in the art.

Page 3 of the official action argues that “The claims do not reflect the test conditions referred to by appellants’ arguments and declaration.” Appellants note that claims 1 and 16 each recite

“the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap”

wherein, Dr. Freidank concludes that the phrase “that the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap” is clear for the person skilled in the art. Any differences between the claim language and the term opined to by Dr. Freidank are insignificant.

While the examiner has expressed his confusion as to the meaning of the term based on his inability to determine when a peak begins or ends, such confusion does not render the

claim indefinite, as Dr. Freidank has opined that the term is clear for the person skilled in the art. The Freidank declaration is offered as **evidence** that the claim term is not indefinite.

The examiner acknowledges the usefulness of the Freidank Declaration in determining the distribution in valleys between peaks, but erroneously dismisses the declaration as not addressing the examiner's concerns as to what particle sizes are included or excluded by the claims. Pgs 2 and 3 of official action.

*Degree Of Overlap Is Not Necessary To Understand The Metes And Bounds Of The Claims*

The examiner erroneously concludes that it is necessary to be able to determine what distribution of particle sizes are included or excluded in order to understand the metes and bounds of the claims, and that such a determination is not possible in the absence of understanding when a peak begins and ends.

The examiner has erroneously adopted a meaningless framework for analysis and concluded that since the framework can not be addressed, the claim is indefinite.

The simple matter to be decided by the board is whether the metes and bounds of the term **a bimodal particle size distribution and the peaks of the large and small particles...do not overlap** is clear to those of ordinary skill in the art.

The examiner has not concluded that the metes and bounds of "bimodal particle size distribution" is indefinite. Accordingly, the only issue is to determine, in the context of a bimodal distribution, the meaning of "do not overlap."

The evidence of record (Freidank declaration) is that "do not overlap" is that data measured **between peaks** falls to a degree which is below background-noise (e.g. no signal detected). The examiner has not rebutted such a conclusion. The examiner's criticism is



merely that the declaration fails to address the examiner's curiosity as to what particle size distribution are included or excluded.

Quite simply if the particle size distribution is bimodal and the peaks do not overlap, the distribution is included. If the particle size distribution is not bimodal the distribution is excluded. If the particle size distribution includes peaks which overlap, the distribution is excluded. Determination of when a peak begins and ends is not necessary provided one can determine whether there is overlap or not. Accordingly, the metes and bounds of the claims are clear.

Furthermore, the examiner has provided no reasoning or evidence as to why it is necessary to determine when a peak begins or ends in order to understand the metes and bounds of the claims.

Thus, the examiner has committed reversible error by substituting his own understanding which is in contradiction to the opinion of one of ordinary skill in the art.

The examiner's rejection under 35 U.S.C. 112, § second paragraph is improper and must be reversed.

The examiner has committed reversible error in concluding the claimed invention to be obvious over the cited references as none of the cited art of record discloses or suggests a graft polyol having **a bimodal particle size distribution**.

The examiner has rejected claims 1, 2 and 4-17 under 35 U.S.C. § 103(a) over EP 786,480 in view of Perry et al. (U.S. 6,127,443).

*No Disclosure of A Bimodal Particle Size Distribution Where The Peaks Do Not Overlap*

None of the cited art of record discloses or suggests a graft polyol having a bimodal particle size distribution **in which the peaks of the large and small particles do not overlap.**

EP '480 merely describes a **polymer polyol** having a relatively small and having a **narrow particle size distribution** (page 1, lines 3-5 and page 3, lines 2-7). In describing a polymer polyol having a small particle size and a **narrow particle size distribution**, there is no suggestion of a graft polyol having a **bimodal particle size distribution** in which the peaks do not overlap. A bimodal particle size distribution is not a narrow particle size distribution. To the contrary, a bimodal distribution is quite contrary to a narrow particle size distribution.

Perry et al. merely describes a polyol component which is **at least bicompositional** having at least one high **molecular weight** portion and one low **molecular weight** portion (column 3, lines 17-19). The molecular weight is a characterization of the length of the polymer chains of the polymer and says **nothing about the particle size of polymer particles.** There is no disclosure in this reference as to a bimodal **particle size** distribution in which the peaks do not overlap.

A recitation of a **bicompositional** composition is not a suggestion of a **bimodal** particle size distribution. The term bicompositional refers to the qualitative nature of the composition components such that there are components of **two different compositions.** A bimodal particle size describes the average particle size of the particles of the composition such that there are two peaks, describing the **particle size distribution.** Differences in composition do not suggest differences in particle size distribution.

Moreover, even if Perry et al. were to have described a bimodal particle size distribution, there is no motivation to modify the polymer polyol of EP '480 to provide a bimodal distribution as to do so would be contrary to the express teachings of EP '480.

EP '480 describes a polymer polyol having a small particle size and **a narrow particle size distribution**. A narrow particle size distribution is a statement as to the desirability of uniform properties for the polymer particles. A bimodal particle size is inconsistent with a narrow particle size distribution as a bimodal particle size has two particle size distributions and therefore is nearly the opposite of a narrow particles size distribution. It would not be possible to modify the disclosure of EP '480 and provide a bimodal particle size distribution as to do so would destroy the essential teachings of the primary references. Obvious modifications can not fly in the face of the express disclosure of the reference. As such the combination of cited references does not make obvious a graft polyol having a bimodal particle size distribution.

In contrast, the claimed invention is directed to a graft polyol having small and large particles having a bimodal particle size distribution in which the peaks of the large and small particles do not overlap.

The paragraph bridging pages 7-8 of the official action "holds" but without any evidence, that a bicompositional composition from two different components with each component having different and independent narrow particle size distributions, was within the level of skill of those of ordinary skill in the art, from operating within the teachings of the combined prior art in order to arrive at the products and processes of appellants' claims.

Such a holding insufficient to establish a *prima facie* case of obviousness.

A statement that modifications of the prior art to meet the claimed invention would have been "well within the ordinary skill of the art at the time the claimed invention was made" because the references relied upon teach that all aspects of the claimed invention were individually known in the art is not sufficient to establish a *prima facie* case of obviousness without some objective reason to combine the teachings of the references. *Ex parte Levengood*, 28 USPQ2d 1300 (Bd. Pat. App. & Inter. 1993).

\*\*\*">[R]ejections on obviousness cannot be sustained by mere conclusory statements; instead, there must be some articulated reasoning with some rational underpinning to support the legal conclusion of obviousness.'" *KSR*, 550 U.S. at \_\_\_, 82 USPQ2d at 1396 quoting *In re Kahn*, 441 F.3d 977, 988, 78 USPQ2d 1329, 1336 (Fed. Cir. 2006).<M.P.E.P. § 2143.01 IV

Not only is such a holding insufficient to establish a prima facie case of obviousness, but such a holding is simply not supported by the evidence of record.

While the examiner asserts that a blend of polymers is suggestive of a bimodal particle size distribution, appellants note that the secondary reference suggests the use of two polymer **compositions**, **not** two polymers having different **particle size distributions**. Moreover by combining the teachings of the two references, one would use two polymers of **the same particle size distribution**, providing a bicompositional polyol having **a narrow particle size distribution**.

Further more, a narrow particle size distribution is demonstrated in EP '460 as a factor in providing a desired low viscosity. Table 1, on page 7 demonstrates a significantly better viscosity when the particle size span is only 0.85  $\mu\text{m}$  (example 1) as compared to when the particle size span is 1.74  $\mu\text{m}$  (Comp. Ex. 1). Such a dichotomy is also demonstrated in Table 2, page 8 in which a significantly better viscosity when the particle size span is only 0.88  $\mu\text{m}$  (example 2) as compared to when the particle size span is 1.41  $\mu\text{m}$  (Comp. Ex.2). Thus, deviations from a narrow particle size distribution has been demonstrated to be less desirable than a narrow particle size distribution. Clearly the combination of two different particle size distributions would produce a composition having a broader particle size distribution than the described "narrow particle size distribution" of EP '480. Such a combination is contrary to the essential disclosure of EP '460 in providing a low viscosity composition.

If proposed modification would render the prior art invention being modified unsatisfactory for its intended purpose, then there is no suggestion or motivation to

make the proposed modification. *In re Gordon*, 733 F.2d 900, 221 USPQ 1125 (Fed. Cir. 1984)

The evidence of record contradicts the examiner's conclusion as to motivation to combine the teachings for the two references.

Even if one were to combine the disclosures of the cited references there is still no suggestion of providing a polyol in which the peaks of the large and small particles **do not overlap**. At best the office action has provided references which describe a narrow particle size distribution as well as a bicompositional composition. The combined disclosure would suggest a composition in which the particles for the two compositions would overlap completely, resulting in a narrow particle size distribution. As noted above, a narrow particle size distribution was demonstrated as essential to providing uniformity and a low viscosity. None the less, assuming that there were any suggestion to have two particle size distributions, there is no suggestion that the peaks of the two distributions would **not overlap**. There is simply no evidence cited in the official action to provide two distributions in which the peaks do not overlap.

As the combined teachings of the cited prior art fails to disclose or suggest a bimodal particle size distribution in which the peaks do not overlap, the claimed invention is clearly not obvious from these references.

In view of the errors made by the examiner, his conclusion as to obviousness is in error and must be reversed.

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Respectfully submitted,

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(viii) Claims Appendix

Claim 1 A graft polyol comprising  
small particles having a particle diameter of from 0.05 to 0.7  $\mu\text{m}$  and  
large particles having a particle diameter of 0.4 to 5.0  $\mu\text{m}$ , wherein said large particles  
have a larger particle size than said small particles, and  
wherein the graft polyol has a bimodal particle size distribution,  
the peaks of the large and small particles measured by the Fraunhofer diffraction  
method in combination with polarization intensity differential scattering do not overlap, and  
the graft polyol has a total solids content of from 5 to 65% by weight  
wherein the total content of the solids consists of a volume fraction of from 5 to 45%  
of the small particles and a volume fraction of from 95 to 55% of the large particles, wherein  
the volume fractions of the small and large particles sum up to 100%.

Claim 2 The graft polyol as claimed in claim 1, wherein the peak of the small  
particles, measured by the Fraunhofer diffraction method in combination with polarization  
intensity differential scattering, begins in a range of from 0.05 to 0.08  $\mu\text{m}$  and ends in a range  
of from 0.4 to 0.7  $\mu\text{m}$  and the peak of the large particles, measured by the Fraunhofer  
diffraction method in combination with polarization intensity differential scattering, begins in  
a range of from 0.4 to 1.0  $\mu\text{m}$  and ends in a range of from 1.2 to 5.0  $\mu\text{m}$ .

Claim 4 The graft polyol as claimed in claim 1, wherein the small particles have a  
diameter of from 0.1 to 0.5  $\mu\text{m}$  and the large particles have a diameter of from 0.5 to 4.0  $\mu\text{m}$ .

Claim 5 The graft polyol as claimed in claim 1, wherein the total solids content of the  
graft polyol is from 10 to 50% by weight.

Claim 6 The graft polyol as claimed in claim 1, wherein the total content of the solids consists of a volume fraction of from 10 to 40% by weight of the small particles and a volume fraction of from 90 to 60% by weight of the large particles, wherein the volume fractions of the small and large particles sum up to 100%.

Claim 7 A process for the preparation of the graft polyols as claimed in claim 1 comprising:

mixing (i) at least one graft polyol having a monomodal particle size distribution with small particles which have a diameter of from 0.05 to 0.7  $\mu\text{m}$  with (ii) at least one graft polyol having a monomodal particle size distribution with large particles which have a diameter of from 0.4 to 5.0  $\mu\text{m}$  to form a graft polyol having a bimodal particle size distribution wherein the total solids content of the graft polyol having a bimodal particle size distribution consists of a volume fraction of from 5 to 45% of small particles and a volume fraction of from 95 to 55% of large particles, wherein the volume fractions of the small and large particles sum up to 100%.

Claim 8 The process as claimed in claim 7, wherein the small particles have a particle diameter of from 0.1 to 0.5  $\mu\text{m}$ .

Claim 9 The process as claimed in claim 7, wherein the large particles have a particle diameter of from 0.5 to 4.0  $\mu\text{m}$ .

Claim 10 The process as claimed in claim 7, wherein the graft polyol having a bimodal particle size distribution has from 10 to 40% by volume of the graft polyol having a



monomodal particle size distribution with small particles and from 90 to 60% of the graft polyol having a monomodal particle size distribution of large particles, wherein the volume fractions of the graft polyols having a monomodal particle size distribution with small and large particles sum up to 100%.

Claim 11 A process for the preparation of a graft polyol having a bimodal particle size distribution comprising

small particles having a particle diameter of from 0.05 to 0.7  $\mu\text{m}$  and

large particles having a particle diameter of 0.4 to 5.0  $\mu\text{m}$ , wherein said large particles have a larger particle size than said small particles, and

wherein the graft polyol has a bimodal particle size distribution,

the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap, and

the graft polyol has a total solids content of from 5 to 65% by weight

wherein the total content of the solids consists of a volume fraction of from 5 to 45% of the small particles and a volume fraction of from 95 to 55% of the large particles, wherein the volume fractions of the small and large particles sum up to 100%

comprising preparing graft polyols in a semibatch process, wherein the initially taken reaction mixture contains in each case at least one carrier polyol, a macromer and a graft polyol having a monomodal particle size distribution, more than 3% by weight of the solids content in the resulting graft polyol consisting of the solids content of the graft polyol used in the initially taken reaction mixture and having a monomodal particle size distribution, and the amount of the macromer used in the initially taken reaction mixture is from 1 to 30% by weight, based on the total weight of the ethylenically unsaturated monomers wherein the amount is sufficient to form small particles in the further course of the reaction.

Claim 12 A process as claimed in claim 11, wherein the amount of macromer used in the initially taken reaction mixture is from 2 to 15% by weight, based on the amount of the ethylenically unsaturated monomers.

Claim 13 A process as claimed in claim 11, wherein the macromer is a polyol having an average molecular weight of more than 2 000 g/mol and a functionality of  $\geq 2$ , wherein said macromer has at least one terminal, polymerizable, ethylenically unsaturated group.

Claim 14 A process as claimed in claim 13, wherein the macromer is a polyol having an average molecular weight of more than 3 000 g/mol.

Claim 15 A polyurethane which comprises the graft polyol as claimed in claim 1.

Claim 16 A process for the preparation of polyurethanes comprising reacting (a) organic and/or modified organic polyisocyanates with (b) graft polyols and, optionally (c) additional compounds having hydrogen atoms reactive toward isocyanates, in the presence of (d) catalysts, (e) optionally water and/or other blowing agents and, optionally (f) additional assistants and additives,

wherein the (b) graft polyols have a bimodal particle size distribution and a total solids content of from 5 to 65% by weight, which comprises small particles having a diameter of from 0.05 to 0.7  $\mu\text{m}$  and large particles having a diameter of from 0.4 to 5.0  $\mu\text{m}$  wherein said large particles have a larger particle size than said small particles,

the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap,

and the total content of solids consists of a volume fraction of from 5 to 45% of the small particles and a volume fraction of from 95 to 55% of the large particles, wherein the volume fractions of the small and large particles sum up to 100%.

(ix) Evidence Appendix

Daniel Freidank declaration submitted July 26, 2007

Daniel Freidank declaration submitted February 26, 2008

Daniel Freidank declaration submitted August 19, 2008

## IN THE UNITED STATES PATENT &amp; TRADEMARK OFFICE

In re Application of

Bernd Zschke et al.

Serial Number 10,507,315

Filed: 09/10/2004

For: GRAFT POLYOLS WITH A BIMODAL PARTICLE SIZE DISTRIBUTION AND  
METHOD FOR PRODUCING GRAFT POLYOLS OF THIS TYPE, IN ADDITION TO  
THE USE THEREOF FOR PRODUCING POLYURETHANES

## DECLARATION UNDER 37 CFR 1.132

I, Daniel Freidank, a citizen of the Federal Republic of Germany and residing at  
67063 Ludwigshafen, Federal Republic of Germany, declare as follows:

I am a fully trained chemist, having studied chemistry at the University of Bielefeld, 33501  
Bielefeld, Federal Republic of Germany, from 1993 to 1999 and at the University of Freiburg,  
79110 Freiburg i. Br., Federal Republic of Germany, from 2000 to 2003.

I received my Doctors degree at the University of Freiburg in 2005,

I joined BASF AG, 67056 Ludwigshafen, Federal Republic of Germany, in 2003, since when  
I have been working in the field of polyurethane research and development,

I am well acquainted with technical English,

To demonstrate that the graft polyols prepared according to the above patent application have a bimodal particle size distributions with two separate peaks, that do not overlap, I made the following examples.

### Comparative Experiment

Six samples of graft polyols had been prepared in accordance to Example 4 of the above patent application.

The particle size distribution of the graft polyols was measured by "laser diffraction analysis" in combination with "polarization intensity differential scattering (PIDS)". This method for the determination of particle size distribution is an important standard method in order to characterize dispersions. The companies Coulter and Malvern are the two major suppliers of instruments for particle size analysis based on laser diffraction.

The particle size distribution of a graft polyol sample according to the claims of WO 03/078496 was determined with a Laser Diffraction Particle Size Analyzer from Beckman Coulter GmbH Krefeld ([www.beckmancoulter.com](http://www.beckmancoulter.com)) under the following conditions:

#### Instrument:

|                        |   |
|------------------------|---|
| Name:                  | Laser Diffraction Particle Size Analyzer LS 230   |
| Dynamic Size Range:    | 0,04 $\mu\text{m}$ up to 2000 $\mu\text{m}$   |
| Measurement Principle: | Fraunhofer theory of light scattering in combination with Polarization Intensity Differential Scattering (PIDS)   |
| Laser wavelength:      | 750 nm  |
| PIDS light source:     | Tungsten-Halogen-lamp<br>3 filters for polarization in the vertical and horizontal planes (450 nm; 600 nm; 900 nm)<br>Measurement of light scattering at six scattering angles (60°; 75°; 90°; 105°; 120° and 146°) |
| Dispersion unit:       | Small Volume Module (SVM-Module)  |
| Detector:              | 132 detectors; 116 size channels  |
| Software:              | LS32 Version 3.01 (Microsoft Windows based)   |

#### Evaluation:

|   |  |
|---|--|
| Optical model:  | PSL (Polystyren Latex) with PIDS; Formfactor 1 |
| Measurement liquid:                                   | 2-Propanol                                     |
| Liquid refraction index:                              | 1,374  |
| Real part of refraction index of the sample:          | 1,6  |
| Imaginary part of the refraction index of the sample: | 0,0  |

#### Sample preparation:

0,5 ml Graftpolyol are added with a Pasteur pipette to 15 ml Isopropanol (2-Propanol) in a 30 ml-beaker. The components are well mixed. The dispersion is introduced drop wise into the dispersion unit of the measurement instrument. Sample introduction is finished when the instrument signalizes adequate density. Then the measurement can be started.

The results of the measurement are displayed graphically and as table

**Picture 1**

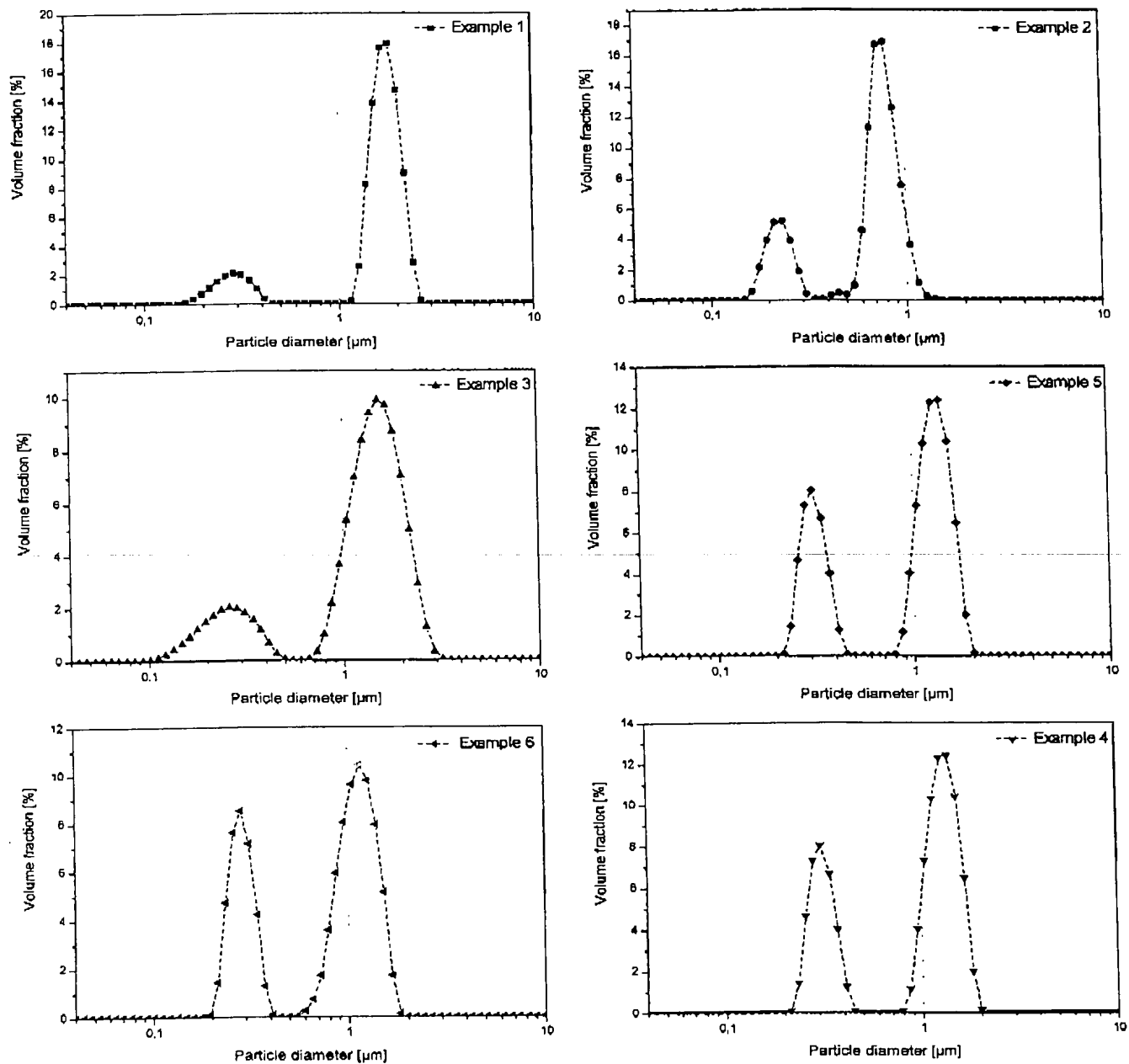


Table 1 (numerical data of examples 1-6):

| Particle diameter<br>[µm] | Example 1              |       | Example 2              |       | Example 3              |       | Example 4              |       | Example 5              |       | Example 6              |       |
|---------------------------|------------------------|-------|------------------------|-------|------------------------|-------|------------------------|-------|------------------------|-------|------------------------|-------|
|                           | Volume fraction<br>[%] | Error | Volume fraction<br>[%] | Error | Volume fraction<br>[%] | Error | Volume fraction<br>[%] | Error | Volume fraction<br>[%] | Error | Volume fraction<br>[%] | Error |
| 0,040                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,044                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,048                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,053                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,058                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,064                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,070                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,077                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,084                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,093                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,004 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,102                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,027 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,112                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,098 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,122                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,230 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,134                     | 0,001 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,420 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,148                     | 0,015 +/- 0,179        |       | 0,037 +/- 0,169        |       | 0,640 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,162                     | 0,100 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,890 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,176                     | 0,330 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,195                     | 0,390 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,214                     | 0,430 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,235                     | 0,580 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,258                     | 0,990 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,284                     | 2,120 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,311                     | 2,020 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,342                     | 0,330 +/- 0,179        |       | 0,023 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,375                     | 0,040 +/- 0,179        |       | 0,029 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,412                     | 0,030 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,452                     | 0,029 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,970 +/- 0,099        |       | 0,061 +/- 0,124        |       | 0,100 +/- 0,124        |       | 0,100 +/- 0,104        |       |
| 0,496                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,048 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,545                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,004 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,027 +/- 0,104        |       |
| 0,598                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,003 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,657                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,050 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,721                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,030 +/- 0,099        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,791                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,869                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 0,953                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,047                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,149                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,261                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,385                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,520                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,669                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 1,832                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 2,010                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 2,207                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 2,423                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 2,660                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 2,920                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 3,208                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 3,519                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 3,862                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 4,241                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 4,656                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 5,111                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 5,611                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 6,158                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 6,781                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 7,421                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 8,147                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 8,944                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 9,818                     | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 10,780                    | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |
| 11,830                    | 0,000 +/- 0,179        |       | 0,000 +/- 0,169        |       | 0,000 +/- 0,099        |       | 0,100 +/- 0,124        |       | 0,078 +/- 0,124        |       | 0,000 +/- 0,104        |       |

For better visualization, those datapoints, which contribute to the peaks of the comparative examples are highlighted with grey background. The manufacturer of the instruments states a reproducibility of 1%. Therefore a data point is regarded to contribute to the peak of the particle size distribution only, if its value is larger than the typical relative error (1%) of the peak maximum.

It can be stated, that all comparative examples are in accordance with the claims WO 03/078496. The peaks are well separated and do not overlap.



I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at 67056 Ludwigshafen, Germany, this

day of July 13<sup>th</sup> 2007

David Frolck  
Signature of Declarant

## IN THE UNITED STATES PATENT &amp; TRADEMARK OFFICE

In re Application of  
Bernd Zschke et al.  
Serial Number 10,507,315  
Filed: 09/10/2004

For: GRAFT POLYOLS WITH A BIMODAL PARTICLE SIZE DISTRIBUTION AND  
METHOD FOR PRODUCING GRAFT POLYOLS OF THIS TYPE, IN ADDITION TO  
THE USE THEREOF FOR PRODUCING POLYURETHANES

## DECLARATION UNDER 37 CFR 1.132

I, Daniel Freidank, a citizen of the Federal Republic of Germany and residing at  
67063 Ludwigshafen, Federal Republic of Germany, declare as follows:

I am a fully trained chemist, having studied chemistry at the University of Bielefeld, 33501  
Bielefeld, Federal Republic of Germany, from 1993 to 1999 and at the University of Freiburg,  
79110 Freiburg i. Br., Federal Republic of Germany, from 2000 to 2003.

I received my Doctors degree at the University of Freiburg in 2005,

I joined BASF AG, 67056 Ludwigshafen, Federal Republic of Germany, in 2003, since when  
I have been working in the field of polyurethane research and development,

I am well acquainted with technical English,

To demonstrate the meaning of the phrase "that the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap" I declare the follow.

I refer to the comparative examples of my Declaration under 37 CFR 1.132 of July 19, 2007.

It had been shown in the Declaration of July 19, 2007 that the diagrams resulting from the results of the measurements of the particle size distribution show two peaks which are separated from each other so that there was no overlap of the peaks.

As every measurement method the polarization intensity differential scattering has a so called background noise of the measurement.

The background-noise of the measurement is in the order of magnitude of typically 1% of the peak maximum. Therefore data-points, the value of which is less than this, are not a signal, but part of the indefinite background. Therefore they cannot be distinguished from "nothing", and it can be stated, that the peaks do not overlap.

In the current patent application it means that data measured between the peaks are below the background-noise, or, with other words, between the peaks is an area of measurements not larger as the background-noise.

The polarization intensity differential scattering is an established method for determining the particle size in the range of the graft polyols claimed in the patent application Serial Number 10,507,315.

The Laser Diffraction Particle Size Analyzer LS 230 used for the Comparative Examples in my Declaration of July 19, 2007 is common used for these measurements.

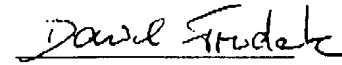
So art the meaning of the phrase "that the peaks of the large and small particles measured by the Fraunhofer diffraction method in combination with polarization intensity differential scattering do not overlap" is clear for the person skilled in the art.

It can be stated, that all comparative examples are in accordance with the claims WO 03/078496. The peaks are well separated and do not overlap.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at 67056 Ludwigshafen, Germany, this

day of

  
Signature of Declarant

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In re Application of

Bernd Zäschke et al.

Serial Number 10,507,315

Filed: 09/10/2004

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METHOD FOR PRODUCING GRAFT POLYOLS OF THIS TYPE, IN ADDITION TO  
THE USE THEREOF FOR PRODUCING POLYURETHANES

D E C L A R A T I O N   U N D E R 37 CFR 1.132

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Signed at 67056 Ludwigshafen, Germany, this

12 day of August, 2008

David Fridale

Signature of Declarant

(x) *Related Proceedings Appendix*

none